Analysis of Soil and Sludges Using SW-846 Method 6010c on the Teledyne Leeman Labs Dual-View Prodigy Plus ICP-OES

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Introduction

This application note demonstrates the capability of the Teledyne Leeman Labs Prodigy Plus High Dispersion ICP to perform analysis of soils and sludges according to SW-846 Method 6010c. This method is applicable to ground waters, toxicity characteristic leaching procedure (TCLP), extraction procedure (EP) toxicity extracts, industrial organic wastes, soils, sludge and sediments.

Instrumentation

A Prodigy Plus High-Dispersion ICP system equipped with a dual-view torch and 120-position CETAC ASX-280 autosampler (Figure 1) was used to generate the data for this application note.

Figure 1 Prodigy Plus ICP-OES and ASX-280 Autosampler

The Prodigy Plus is a compact benchtop simultaneous ICP-OES system featuring an 800 mm focal length Echelle optical system coupled with a mega-pixel large format CMOS detector. At 28 x 28 mm, the active area of the CMOS detector is significantly larger than any other solid-state detector currently used for ICP-OES. This combination allows the Prodigy Plus to achieve higher optical resolution and dispersion than any other solid-state detector-based ICP systems. The detector also provides continuous wavelength coverage from 165 to 1100 nm, permitting measurement over the entire ICP spectrum in a single reading, without sacrificing wavelength range or resolution. The detector design is inherently anti-blooming and is capable of random access, non-destructive readout that results in a dynamic range of more than six orders of magnitude. The Prodigy Plus uses a 40.68 MHz rugged, free-running RF generator, allowing it to handle the most difficult sample matrices, as well as common organic solvents.

Sample Introduction

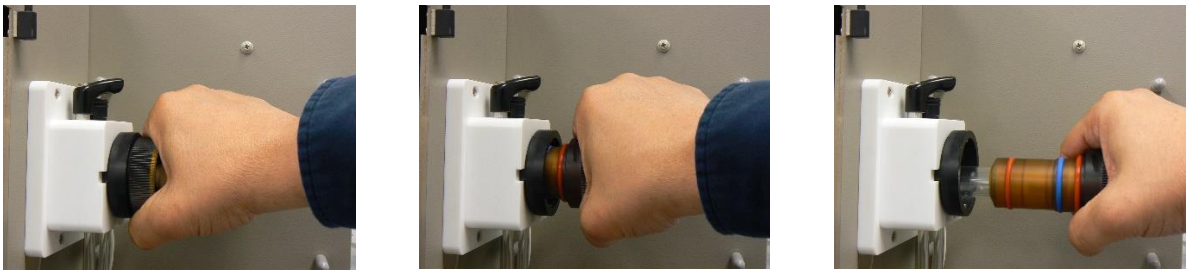
A high-sensitivity sample introduction system ensured that sufficient and steady emission signals were delivered to the spectrometer. The sample introduction system consisted of:

- Cyclonic Spray Chamber with Center Knockout Tube
- Seaspray™ High Solids AR30 Concentric Nebulizer
- Four-Channel Peristaltic Pump

The volume of the cyclonic spray chamber is low allowing for fast washout between samples, while its knockout tube (or baffle) efficiently reduces the amount of sample aerosol that reaches the torch.

The Prodigy Plus torch is mounted using an innovative twist-lock auto-aligning sample introduction system, shown in Figure 2. This design permits operators to remove and replace the torch to the exact same position, providing day-to-day reproducibility and simplified training. Additionally, the twist-lock design automatically connects the coolant and auxiliary gas flows, eliminating potential errors.

Figure 2 Axial Twist Lock Sample Introduction System



Method

The Prodigy Plus instrument operating parameters are shown in Table I.

Table I Instrument Operating Parameters

Parameter	Value	Part Number
RF Power	1.2 kW	-
Coolant Flow	12 L/min	-
Auxiliary Flow	0.8 L/min	-
Plasma Configuration	Dual View (Axial and Radial)	-
Nebulizer Pressure	30 psi	-
Pump Rate	30 rpm	-
Torch	Demountable Quartz (Axial, Radial and Dual)	318-00167-1
Injector	2.5 mm Bore Quartz Demountable	318-00161-AQ1
Sample Uptake Time	35 s	-
Replicates	3	-
Nebulizer	Seaspray™ High Solids AR30 Concentric	120-00474-1
Spray Chamber	Cyclonic with Knock-out	120-00393-1
Rinse Time	20 s	-
Integration Time	Axial 30 s, Radial 15 s	-
Optical Purge Rate	0.7 L/min	-
QC Limit	±10%	-
QC Failure Action	Recalibrate and Rerun	-

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Wavelengths

Element wavelengths and plasma viewing mode used are shown in Table II.

Table II Wavelengths		
Element	**Wavelength**	**View**
Ag	328.068	Axial
As	193.759	Axial
Ba	233.527	Axial
Be	313.042	Axial
Cd	214.441	Axial
Co	228.615	Axial
Cr	267.716	Axial
Cu	324.754	Axial
Mn	257.61	Axial
Ni	231.604	Axial
Pb	220.353	Axial
Sb	206.833	Axial
Se	196.09	Axial
Tl	190.856	Axial
V	309.311	Axial
Zn	213.856	Axial
Al	308.215 r	Radial
Ca	315.887 r	Radial
Fe	259.940 r	Radial
K	766.491 r	Radial
Mg	279.078 r	Radial
Na	589.592 r	Radial

Calibration Standards

Calibration standards were prepared from multi-element stock solutions in appropriate concentrations to cover the linear range for each element. The acid concentration of the standards was carefully matched to the acid concentration of the prepared soil samples. The suggested element mix in Method 6010c was used, and is shown in Table III.

Table III Calibration Standard Concentration, mg/mL						
Elements	**STD1**	**STD2**	**STD3**	**STD4**	**STD5**	**STD6**
Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sb, Se, Tl, V, Zn	0	1	5	10	-	-
Al, Ca, Fe, K, Mg, Na	0	-	-	-	10	100

Quality Control (QC)/Check Standards

The initial calibration verification (ICV) standard was prepared by combining compatible elements from a standard source different from that of the calibration standard, and at a concentration near the midpoint of the calibration curve. A purchased second-source reference standard can also be used.

The continuing calibration verification (CCV) standard was prepared in the same acid matrix, using the same standards as those used for calibration standards and at a concentration near the midpoint of the calibration curve. The Prodigy Plus Salsa software was set to recalibrate and rerun all samples measured since the last successful CCV, should the CCV fail.

Results

ICV and CCV Calibration Results

Typical results from the ICV and CCV are shown in Table IV and Table V respectively.

Table IV Typical ICV Results

Element	Actual	Measured	%Recovery
Ag	5.00	4.77	95.5
As	5.00	4.90	98.0
Ba	5.00	5.25	105.1
Be	5.00	5.27	105.5
Cd	5.00	4.61	92.2
Co	5.00	4.71	94.3
Cr	5.00	4.81	96.2
Cu	5.00	5.08	101.5
Mn	5.00	4.64	92.7
Ni	5.00	5.04	100.8
Pb	5.00	4.82	96.4
Sb	5.00	5.23	104.5
Se	5.00	4.51	90.2
Tl	5.00	4.64	92.7
V	5.00	4.52	90.3
Zn	5.00	4.56	91.2
Al	5.00	5.34	106.9
Ca	5.00	5.30	106.0
Fe	5.00	5.23	104.5
K	50.00	51.39	102.8
Mg	5.00	4.63	92.5
Na	5.00	4.60	92.0

Table V Typical CCV Results			
Element	**Actual**	**Measured**	**%Recovery**
Ag	5.00	4.83	96.6
As	5.00	5.14	102.8
Ba	5.00	5.03	100.7
Be	5.00	4.48	89.7
Cd	5.00	4.66	93.1
Co	5.00	4.99	99.9
Cr	5.00	5.16	103.2
Cu	5.00	5.12	102.5
Mn	5.00	4.67	93.4
Ni	5.00	4.57	91.3
Pb	5.00	4.99	99.9
Sb	5.00	4.58	91.5
Se	5.00	4.63	92.5
Tl	5.00	4.31	86.1
V	5.00	4.89	97.8
Zn	5.00	5.05	101.0
Al	50.0	49.50	99.0
Ca	50.0	48.79	97.6
Fe	50.0	54.18	108.4
K	50.0	48.02	96.0
Mg	50.0	51.29	102.6
Na	50.0	54.39	108.8

Sample Results

An ERA WasteWatR™ Pollution Trace Metals (Lot P213-500) certified water sample along with four certified soil samples: ERA Metals in SewageSludG™ (Lot. D078-160) (Environmental Resource Associates, Arvada, CO.), Standard Reference Materials (SRM) 2709, SRM 2710 and SRM 2710 (NIST, Gaithersburg, MD), were prepared and analyzed. Approximately 1 gram of soil was prepared and diluted to 100 mL according to US EPA Method 3050B. Analytical results for the samples are shown in Table VI through Table X. ND indicates the element was not detected.

Table VI ERA WasteWatR™ Pollution Trace Metals, Lot P213-500					
Element	**Concentration Found, mg/L**	**%RSD**	**Certified Value, mg/L**	**% Recovery**	**QC Performance Acceptance Limits**
Ag	298	0.4	308	96.7	275 - 339
As	438	0.7	491	89.1	423 - 545
Ba	317	0.4	339	93.4	307 - 370
Be	289	0.5	329	87.8	296 - 359
Cd	475	0.5	498	95.4	435 - 533
Co	291	0.4	277	105.1	257 - 307
Cr	310	0.4	319	97.2	285 - 348
Cu	425	0.3	409	104.0	365 - 446
Mn	2037	0.4	1980	102.9	1800 - 2160
Ni	2345	0.5	2420	96.9	2180 - 2660
Pb	1443	0.4	1460	98.8	1300 - 1620
Sb	233	1.5	246	94.8	212 - 276
Se	531	1	486	109.3	422 - 549
Tl	798	0.8	748	106.7	651 - 838
V	255	0.5	287	88.7	262 - 307
Zn	1596	0.3	1540	103.7	1390 - 1710
Al	621	2.6	690	90.0	594 - 794

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Table VII ERA Metals in SewageSludG™, Lot D078-160					
Element	**Concentration Found, mg/L**	**%RSD**	**Certified Value, mg/L**	**% Recovery**	**QC Performance Acceptance Limits**
Ag	80.63	0.5	92.9	86.8	65.4 - 120
As	278	0.5	284	97.7	216 - 351
Ba	929	0.5	1010	91.9	742 - 1270
Be	65.01	0.5	77.1	84.3	65.7 - 88.4
Cd	103.0	0.6	120	85.9	94.6 - 145
Co	30.18	0.5	28.1	107.4	24 - 32.2
Cr	168.5	0.5	165	102.1	136 - 195
Cu	738.0	0.4	867	85.1	738 - 997
Mn	364.1	0.6	411	88.6	303 - 518
Ni	157.6	0.4	182	86.6	146 - 218
Pb	145.5	0.4	146	99.7	114 - 178
Sb	162.2	0.5	186	87.2	124 - 248
Se	157.0	0.7	166	94.6	115 - 217
Tl	162.6	1.1	168	96.8	130 -205
V	120.7	0.5	143	84.4	90.7 - 195
Zn	952.3	0.5	1180	80.7	945 - 1420
Al	6822	0.8	7510	90.8	6340 - 8690
Ca	42098	0.7	34300	122.7	27500 - 41000
Fe	22231	0.6	23300	95.4	18500 -28200
K	1719	1.1	1980	86.8	1380 - 2580
Mg	3365	0.8	4000	84.1	3370 - 4620
Na	1039	0.8	1080	96.2	787 - 1360

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Table VIII San Joaquin Soil NIST 2709 (LCS)					
Element	**Concentration Found, mg/L**	**%RSD**	**Certified Value, mg/L**	**% Recovery**	**QC Performance Acceptance Limits**
As	18.15	1.5	<20	-	-
Ba	381.6	0.3	398	392 - 400	95.9
Cd	0.7820	3.3	<1	-	-
Co	13.73	0.6	12	10.0- 15.0	114
Cr	68.13	0.3	79	60 - 115	86.2
Cu	27.56	0.3	32	26 - 40	86.1
Mn	470.0	0.3	470	360 - 600	100
Ni	73.01	0.3	78	65 - 90	93.6
Pb	13.10	4	13	12.0 - 18.0	101
Sb	4.230	5.6	<10	-	-
Se	ND	-	0.014	-	-
V	54.28	0.4	62	51 - 70	87.6
Zn	95.00	0.5	100	87 - 120	95.0
Al	2.558	0.4	2.6	2.0 - 3.1	98.4
Ca	1.465	0.3	1.5	1.4 - 1.7	97.7
Fe	2.747	0.3	3	2.5 - 3.3	91.6
K	0.3266	0.3	0.32	0.26 - 0.37	102
Mg	1.485	0.4	1.4	1.2 - 1.5	106
Na	0.071	0.3	0.068	0.063 - 0.11	105

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Table IX Montana Soil (Elevated), NIST 2710					
Element	**Concentration Found, mg/L**	**%RSD**	**Certified Value, mg/L**	**% Recovery**	**QC Performance Acceptance Limits**
Ag	23.74	1.3	28	24 - 30	84.8
As	594.7	0.9	590	490 - 600	100.8
Ba	378.6	1	360	300 - 400	105
Cd	22.77	1.7	20	13 - 26	114
Co	7.556	2.6	8.2	6.3 - 12	92.2
Cr	22.78	1.1	19	15 - 23	120
Cu	2918	0.9	2700	2400 - 3400	108
Mn	6801	0.8	7700	6200 - 9000	88.3
Ni	9.65	1	10.1	8.8 - 15	96
Pb	4786	0.9	5100	4300 - 7000	94
Sb	7.72	4.1	7.9	3.4 - 12	98
Se	ND	-	0.002	-	-
Tl	ND	-	0.63	0.5 - 0.76	-
V	43.40	0.9	43	37 - 50	101
Zn	5493	0.7	5900	5200 - 6900	93.1
Al	2.169	0.5	1.8	1.2 - 2.6	121
Ca	0.395	0.5	0.41	0.38 - 0.48	96.4
Fe	3.161	0.4	2.7	2.2 - 3.2	117
K	0.461	0.6	0.45	0.37 - 0.50	102
Mg	0.546	0.4	0.57	0.43 - 0.60	95.8
Na	0.057	0.7	0.054	0.049 - 0.062	105

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Table X Montana Soil (Moderate), NIST 2711					
Element	**Concentration Found, mg/L**	**%RSD**	**Certified Value, mg/L**	**% Recovery**	**QC Performance Acceptance Limits**
Ag	4.8	2.9	4	2.5 - 5.5	120
As	88.6	2.9	90	88 - 110	98.4
Ba	184	0.5	200	170 - 260	92.00
Cd	40.8	0.8	40	32 - 46	102.0
Co	7.79	1.5	8.2	7.0 - 12.0	95.0
Cr	19	0.8	20	15 - 25	95.0
Cu	97.3	0.5	100	91 - 110	97.3
Mn	564	1	490	400 - 620	115.1
Ni	17.1	0.8	16	14 - 20	106.9
Pb	990	0.5	1100	930 - 1500	90.0000
Sb	4.88	4.8	<10	-	-
Se	ND	-	0.009	-	-
V	45.1	0.6	42	34 - 50	107.4
Zn	298	0.6	310	290 - 340	96.1
Al	1.93	0.5	1.8	1.2 - 2.3	107.2
Ca	2.42	0.5	2.1	2.0 - 2.5	115.2
Fe	1.87	0.5	2.2	1.7 - 2.6	85.0
K	0.35	0.8	0.38	0.26 - 0.53	92.1
Mg	0.82	0.5	0.81	0.72 - 0.89	101.2
Na	0.027	0.5	0.026	0.020 - 0.029	103.8

Conclusion

The Prodigy Plus ICP easily accommodated all of the soil samples for analysis. Examination of the data indicates that the results for all elements is well within both the performance acceptance limits (PAL) for the ERA soil and the range specified for the NIST SRM soils. Additionally, the results are in good agreement with the certified values for all samples and demonstrate excellent precision, as indicated by the low relative standard deviation (RSD). Finally, the data confirms that the Prodigy Plus is operating well above the detection limit (DL) and limit of quantitation (LOQ) for the elements determined and that the analysis can be performed with a high degree of confidence. The versatility of unlimited wavelength selection, coupled with the excellent resolution and dispersion of the Prodigy Plus, allowed easy selection of highly sensitive wavelengths.

References

1. "Method 6010C (SW-846): Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 3. [Online] https://19january2017snapshot.epa.gov/sites/production/files/2015-07/documents/epa-6010c.pdf (accessed November 22, 2019).